United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program

SOP No.: PDP-QC-09		Page 1 of 8
Title: Minimum Requirements for Confirmation Using GC/MS-MS		
Revision: 1	Replaces: 04/01/00	Effective: 06/01/02

1. Purpose:

To provide uniform minimum requirements for the operation of a mass spectrometer with MS/MS capabilities.

2. Scope:

This standard operating procedure (SOP) shall be followed by all laboratories conducting pesticide residue studies for PDP, including support laboratories conducting stability or other types of studies that may impact the program.

3. Outline of Procedures:

7.1 Apparatus and Materials
7.2 Reference Spectra
7.3 Minimum Requirements for Confirmation in the MS/MS mode
7.4 Documentation

4. Narrative:

This SOP resulted from individual labs' experience with MS/MS. A set of criteria was developed to provide minimum requirements for analyte confirmation.

5. Definitions:

Collision induced dissociation (CID): Process by which an isolated ion is fragmented, producing an MS/MS spectrum.

Structurally significant ion: Ion with a mass/charge (m/z) ratio which indicates a specific structural grouping formed by the fragmentation of a molecule.

Parent ion:	An abundant, structurally significant ion selected from the full scan spectrum to be isolated and subsequently subjected to collision induced dissociation (CID).
Product ion:	Ions formed when the parent ion undergoes CID.
MS/MS spectrum:	Graphical representation of ion intensity vs. M/Z data at a single point in time produced by an isolated mass undergoing CID.
Target ion:	A structurally significant product/parent ion selected from the reference spectrum, typically the most abundant ion, to be used to generate relative abundance ratios with qualifier ions.
Qualifier ions:	Structurally significant product/parent ions chosen from the reference spectrum to show consistent relative abundances when compared to the target ion.

6. References:

- USDA/AMS PDP Quality Assurance (QA)/Technical Meeting, April 9-11, 2002
- USDA/AMS PDP Quality Assurance Meeting, May 18-20, 1999
- USDA/AMS PDP Mass Spectrometry (MS) Work Group Meeting, March 23-24, 1999
- USDA/AMS SOP No. PDP-QC-06, "Minimum Requirements for Operation of GC/Quadrupole Mass Spectrometer in EI Mode for Confirmation", Revision 2, 09/15/97

7. Specific Procedures to be Followed:

This standard operating procedure (SOP) represents minimum PDP requirements and is presented as a general guideline. Each laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in that laboratory.

7.1 Apparatus and Materials

a. Gas Chromatograph/Mass Spectrometer System

1. A temperature programmable gas chromatograph, complete with all accessories and equipped with a mass spectrometer system capable of performing MS/MS.

2. A capillary column, which promotes good chromatography.

3. A mass spectrometer, i.e., an ion trap or triple quadrupole, which is capable of performing MS/MS: isolating an ion and subsequently causing it to undergo further dissociation.

4. A computer system shall be interfaced to the GC/MS that allows it to acquire and store data of all mass spectra obtained throughout the duration of the chromatographic program.

b. Reagents

The mass spectral analysis shall require the use of known standards of the compounds being confirmed. All standard solutions shall be handled according to PDP SOPs.

7.2 Reference Spectra

a. Tune Reference

The tune program provided by the instrument manufacturer shall be used for instrument tuning. The manufacturer's specification for DFTPP, PFTBA, or other specified compound shall be used to establish proper instrument tune. A hardcopy of the tune result will be archived for future QA review.

b. Standard Reference

1. A reference MS/MS spectrum based on the MS/MS conditions of each standard shall be generated and then archived for future reference.

2. MS/MS conditions for individual compounds shall be documented through a table. This table shall include the following: parent ion, product spectrum, target and qualifier ion(s), and the isolation and excitation parameters required to generated the product spectrum.

7.3 Minimum Requirements of MS/MS Confirmation

a. Generate an air/water check within four hours prior to the start of a sequence.

b. A tune profile shall be generated within four hours prior to the start of a sequence.

c. A matrix blank shall be analyzed with each set of samples.

d. A residue in the sample is determined by satisfying the following criteria:

1. If an internal standard is used, the relative retention time (RRT) of the compound of interest to the internal standard within the reference standard and the RRT of the compound of interest to the internal standard within the sample shall be within 0.01.

2. If an external standard is used, the retention time (RT) of the compound of interest in the standard and the RT of the same compound in the sample shall be ± 0.05 minutes.

3. The MS/MS spectrum of the sample shall match the MS/MS spectrum of the standard injected within the same run and under the same conditions, according to the following criteria.

a. The MS/MS spectrum shall consist of one target ion and one or more qualifier ions. Two or more qualifier ions are preferred; however CID conditions may limit the choice to one.

b. The abundance of the target ions and each qualifier ion, as determined by extracted ion chromatograms, shall be greater than or equal to three times the noise of the sample and the matrix in the RT region of interest.

c. The relative abundances of the qualifier ions to the target ions in the sample shall agree to within "20% (absolute) of the relative abundances in the standard.

d. Dealing with Exceptions - When a compound cannot routinely meet the above criteria due to the nature of the compound or the analytical system, an exception can be requested by the Technical Program Manager and reviewed by the QA officer. The request shall explain the exception and how the confirmation will be accomplished by the GC/MS-MS. All requests shall be documented and the documentation shall be maintained by the laboratory.

7.4 Documentation

The following documents shall be kept as part of confirmation QA/QC:

a. Chromatograms for air or water as a means to check for an air/water leak.

b. Hard copies of tuning profiles, generated prior to sample analyses, of any compound used for tuning (PFTBA, DFTPP, etc.) as well as logs of all instrument maintenance such as source cleaning, replacement of inserts, septums, and columns.

d. Copies of chromatograms and extracted ion chromatograms of samples, standards, matrix blanks, and reagent blanks shall be kept with the raw data pack.

Frances M. Gross *05/15/02*

Reviewed By: Frances M. Gross Date
Presiding Member of PDP QA Committee
New York Department of Agriculture and Markets
Food Laboratory
State Office Campus, Building 7
Albany, New York 12235
(518) 457-5734

Diana Haynes *05/21/02*

Reviewed By: Diana Haynes Date
Quality Assurance Officer, PDP
8609 Sudley Road, Suite 206
Manassas, VA 20110
(703) 330-2300

Martha Lamont *05/21/02*

Approved By: Martha Lamont Date
Director, Monitoring Programs Office
8609 Sudley Road, Suite 206
Manassas, VA 20110
(703) 330-2300

ORIGINAL SIGNATURE PAGE MAINTAINED BY USDA, AMS, S&T, MONITORING PROGRAMS OFFICE
ELECTRONICALLY REPRODUCED SIGNATURES

Revision 1	April 2002	PDP QA/Technical Meeting

- Added definition for CID to section 5
- Added provision for dealing with exceptions to subsection 7.3
- Increased ion ratio criteria to 20% in subsection 7.3.d.3.d